MISCELLANEOUS PAPER NO. 6-922

FIELD AND LABORATORY STUDIES OF THE SULFATE RESISTANCE OF CONCRETE

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U. S. Army Engineer Waterways Experiment Station
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FOREWORD

The manuscript of this paper was prepared at the request of Mr. Robert F. Leggett, Director, Division of Building Research, National Research Council, Ottawa, Canada, for inclusion in the symposium described on page 1 of the manuscript. The paper was reviewed and approved for presentation and publication by the Office, Chief of Engineers, on 21 March 1967.

Col. John R. Oswalt, Jr., CE, was Director of the Waterways Experiment Station during the preparation of this paper. Mr. J. B. Tiffany was Technical Director.

FIELD AND LABORATORY STUDIES OF THE SULFATE RESISTANCE OF CONCRETE*

by
Bryant Mather**

ABSTRACT

The Corps of Engineers, U. S. Army, has been concerned for more than a century and a half with concrete construction in marine environments, both in connection with improvement of harbor works and construction of coastal defenses. In connection with flood control and military construction in the interior of the United States, it has been concerned more recently with sulfate attack on concrete from sources other than the sea. Consequently, it has endeavored to develop an understanding of sulfate resistance through both laboratory and field exposure tests. For approximately 30 years, an exposure station has been maintained on the Atlantic Coast near St. Augustine, Florida, at the mean-tide level. At this station, concrete specimens made in the laboratory have generally demonstrated satisfactory sulfate resistance unless they were made with cements containing more than 12% tricalcium aluminate as calculated from chemical analysis.

Relatively few instances of deterioration of concrete in service in Corps of Engineers structures have been reported. The almost universal use of portland cement meeting the requirements of type II, and thus

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*Prepared for presentation at the Symposium on Concrete Performance--Resistance of Concrete to Sulfate and Other Environmental Conditions, sponsored by the Division of Building Research, National Research Council of Canada, at the 63rd Annual Convention, American Concrete Institute, Toronto, Canada, 7 April 1967.

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containing not more than 8% tricalcium aluminate (C_3A), as calculated from chemical analysis, has contributed to this record. Past and present laboratory studies attempt to define more clearly the factors involved in sulfate attack and sulfate resistance. In connection with studies of portland blast-furnace slag cements, tests of mortar bars containing added sulfate indicated that the expansion of such bars was proportional to the calculated C_3A content of the portland-cement clinker constituent of the cement. When these same cements were used in lean mortar bars exposed to sulfate solution, the ranking of the cements with regard to expansion was quite similar, except in one case, but the expansions observed were in most cases very much smaller. In studies on portland cements, observed expansions of added-sulfate mortar bars at one year ranged from more than 1% to less than 0.1%. These results were generally correlated with the C_3A content of the cement, although there were a few striking anomalies. The imperfection of the correlation between expansion in mortar-bar tests for sulfate resistance with calculated C_3A content reflects differences among cements both in the crystallinity of the C_3A and variable influence of other attributes of the cement, as these affect the permeability and strength and the rate of development of impermeability and strength of the paste. Additional studies are in progress using three cements ranging in calculated C_3A content from 5% to 12%, in mortars of varying cement content and varying water to cement ratios, stored in a 5% sodium sulfate solution. The changes in length, modulus of elasticity, porosity, and strength, with time, of these mortars are being observed in the hope that the influence of factors other than the C_3A content of the cement can be better understood. Other studies having as their object the improved interpretation of the variation in composition and crystallinity of the C_3A in portland-cement clinkers are also going on.

INTRODUCTION

At present, the principal approach to the production of sulfate-resisting concrete is to limit the allowable tricalcium aluminate (C_3A) content of the cement to progressively lower limits as the sulfate concentrations that are expected to come into contact with the concrete in service increase. The Corps of Engineers and the U. S. Bureau of Reclamation, for example, require (1)(7) that, where sulfate concentrations exceed 0.20% as water-soluble SO_4 in soil or 1000 ppm as SO_4 in water, type V (sulfate-resisting) cement will be used; where concentrations are in the range 0.10 to 0.20% in soil or 150 to 1000 ppm in water, type II portland cement or type IS(MS) portland blast-furnace slag cement will be used. Where the concentrations are lower than 0.10% in soil or 150 ppm in water, no special precautions are needed. So far as is known, no significant deterioration of concrete due to sulfate attack has been encountered when these precautions have been taken and the estimated sulfate contents have not been significantly exceeded.

In 1966 the British Standards Institution issued a specification for sulfate-resisting portland cement,(13) in which it is stated that "a considerable degree of sulfate resistance is conferred on portland cement if the tricalcium aluminate is limited to 3-1/2 percent." This may be compared with the limits of 5% and 8% respectively on type V and type II in specifications used in the United States.[(2)(14)]

The limitation on C_3A content of cement involves a calculation of C_3A content based on the results of chemical analysis using the formula:

$$\text{Percent } C_3A = 2.650 \times \text{percent } Al_2O_3 - 1.692 \times \text{percent } Fe_2O_3.$$

CHEMICAL REACTIONS

The chemical reactions involved in the attack on conrete by magnesium sulfate, as provided, for example, by exposure to sea water, have been given[(3)] as follows:

a. The magnesium sulfate reacts with hydrated C_3A:

$$\underset{\text{calcium aluminate hydrate}}{2(3CaO.Al_2O_3.12H_2O)} + \underset{\text{magnesium sulfate}}{3(MgSO_4.7H_2O)} \rightarrow$$

$$\underset{\text{calcium aluminum sulfate hydrate (ettringite)}}{3CaO.Al_2O_3.3CaSO_4.31H_2O} + \underset{\text{aluminum hydroxide}}{2Al(OH)_3} + \underset{\text{magnesium hydroxide}}{3Mg(OH)_2} + 8H_2O$$

b. The magnesium sulfate also reacts with calcium hydroxide:

$$\underset{\text{calcium hydroxide}}{Ca(OH)_2} + MgSO_4.7H_2O \rightarrow \underset{\text{calcium sulfate (gypsum)}}{CaSO_4.2H_2O} + Mg(OH)_2 + 5H_2O$$

c. The gypsum formed in b. or d. also reacts with hydrated C_3A:

$$3CaO.Al_2O_3.12H_2O + 3(CaSO_4.2H_2O) + 13H_2O \rightarrow$$

$$3CaO.Al_2O_3.3CaSO_4.31H_2O$$

d. The magnesium sulfate also reacts with calcium silicate hydrate:

$$\underset{\text{calcium silicate hydrate}}{3CaO.2SiO_2.nH_2O} + 3MgSO_4.7H_2O \rightarrow 3CaSO_4.2H_2O + 3Mg(OH)_2$$

$$+ \underset{\text{silica gel}}{2SiO_2.nH_2O} + 12H_2O$$

e. The magnesium hydroxide formed in a. or b. reacts with the silica gel formed in d.

$$4Mg(OH)_2 + SiO_2.nH_2O \longrightarrow \underset{\text{magnesium silicate hydrate}}{4MgO.SiO_2.8.5H_2O} + n - 4.5H_2O$$

Hansen and Offutt[5] have shown the following reactions of C3A, calcium sulfate (gypsum), and water, depending on which form of calcium aluminum sulfate hydrate is formed:

$$3CaO.Al_2O_3 + 3CaSO_4.2H_2O + 25H_2O \longrightarrow 3CaO.Al_2O_3.3CaSO_4.31H_2O$$

88.8	222.3	450	714.7	(46.4)
1			8	

$$3CaO.Al_2O_3 + CaSO_4.2H_2O + 10H_2O \longrightarrow 3CaO.Al_2O_3.CaSO_4.31H_2O$$

88.8	74.1	180.0	319.6	(23.3)
1			3.6	

The numbers in the first line below the equations are participating volumes. The number in parentheses at the right is the volume added to that on the right side of the equation to make its volume equal the sum of those on the left; thus both of these reactions involve a net reduction in volume of the reactants. The second line of numbers compares the relative volume of anhydrous C3A and the volume of calcium aluminum sulfate formed from it by sulfate reaction. In one case the increase in volume is eightfold; in the other nearly fourfold.

MARINE EXPOSURE

In sulfate attack on conrete in sea water, the sulfate is predominantly magnesium sulfate in a solution that contains a great deal more chloride ion than sulfate ion. Lea[4] has suggested that the presence of the chlorides retards or inhibits the expansion of the concrete by sulfate attack but does not reduce the degree of reaction. He cited work attributing this effect to increased solubility of calcium sulfate and calcium aluminate sulfate in chloride solution.

The Concrete Division, U. S. Army Engineer Waterways Experiment Station, has made many studies directly or indirectly concerned with sulfate-resisting concrete. When the Concrete Division was established in 1946 it assumed jurisdiction over an investigation of the effects of variations in cement composition on the durability of concrete initiated in 1939, involving exposure of concrete specimens to sea water at St. Augustine, Florida. Three specimens were made using each of 51 cements; one specimen was broken before it could be installed. In 1950, after 11 years, only 11 of 152 concrete specimens that were installed had failed[12], and eight of these were made using cements having calculated C_3A contents over 12%. In 1966, 121 specimens were still under test; all have relative dynamic moduli above 98% and relative pulse velocity values (V^2) above 80%. Of the 31 no longer under test, 20 are recorded as "broken in handling" and 11 as having "failed." Eight of the 11 "failed" specimens are accounted for as follows:

Cement	Calculated C_3A, %	
E 58	17	3 specimens failed 1946
E 32	14	3 specimens failed 1948
E 3	13	1 failed 1946; 1 failed 1960

The remaining three "failed" specimens represent one each of groups of three made using cements having 4 to 8% C_3A and perhaps might more accurately have been included in the "broken in handling" category.

TRICALCIUM ALUMINATE CONTENT

In many localities type V portland cement, containing 5% or less C_3A calculated from chemical analysis, is not readily available. The question has been asked whether some cements that, upon chemical analysis, yield values for percentages of Al_2O_3 and Fe_2O_3 that cause the calculated C_3A to exceed 5% might not be as sulfate resistant as others where calculated C_3A content is 5% or less, due to the incorporation of some of the Al_2O_3 in constituents of the cement other than C_3A, in ways not contemplated by the phase equilibria relations upon which the calculations are based. In 1956 a sample of cement was received from the U. S. Army Engineer District, Los Angeles, for study. An attempt was made to compare its C_3A content estimated from X-ray diffraction data with that of other cements that had been studied by the light microscope, chemical analysis, and X-ray diffraction. In the report[8] data were given on 19 cements (table 1). Those cements which by quantitative microscope analysis were found to have C_3A contents lower than 4.8 percent did not give X-ray diffraction peaks at 2.70 A that were sufficiently well developed* to permit quantitative measurement of intensity. Correlation coefficients and Student's t were computed for the relations:

	r	t
a. Micrometric C_3A vs X-ray intensity	+0.905	6.65
b. Calculated C_3A vs X-ray intensity	+0.845	5.04
c. Micrometric C_3A vs calculated C_3A	+0.827	5.98

The cement submitted for test did not have a peak at 2.70 A that was well enough developed to scale; it had 3.9% Al_2O_3 and 2.6% Fe_2O_3 and hence

*X-ray diffraction equipment that became available later permits quantitative measurement of C_3A contents below 4.8 percent as indicated by microscope.

6% calculated C_3A. It was concluded that, had it been examined as clinker by microscope methods, it would have been found to contain less than 5% C_3A. It was assumed that the 5% limit on C_3A applicable to type V cements was intended to refer to C_3A that could, under optimum conditions of crystallization of the cement clinker, be found by microscope examination, if such examination were made. It was therefore concluded that the potential sulfate-resisting qualities of this cement would be expected to be as great as required by the provisions of the specifications for type V cement.

In 1957, ten cement samples were received from the U. S. Army Engineer District, Omaha. Data on these cements are given in table 2.[9] Only one of these cements gave a C_3A peak well enough developed to scale; the intensity was 175 counts/sec above background. Four of the cements gave interfering peaks at about 2.72 A, the significance of which has been discussed elsewhere.[10] Four other cements gave detectable indications of the presence of C_3A. Mortar bars made with sufficient added sulfate to give 7% SO_3 by weight of the cement, a quantity sufficient to react with 7.9% C_3A to form high-sulfate calcium aluminum sulfate, were exposed immersed in water for one year, measured for length change, and then examined. Only very minor amounts of calcium aluminum sulfate were found. The expansions observed (table 2) correlate with the MgO content of the cement. It was concluded that all of these cements would be expected to provide sulfate resistance to the extent to be expected of cements meeting the requirements for type V.

LITERATURE REVIEW

From a partial review of the literature [6] completed in 1961 as a basis for planning further studies, it was concluded that (a) no cement or concrete can be totally resistant to sulfate attack under all possible conditions; (b) The degree of sulfate attack depends upon: (1) the type of cement used; (2) the quality of the concrete; (3) sulfate concentration in contact with the concrete; (4) surface protection of the concrete.

PORTLAND BLAST-FURNACE SLAG CEMENTS

Blended cements have not been studied with respect to sulfate resistance to the extent that portland cements have, even though reports have been available in increasing numbers for over 100 years that indicate that most of the materials used together with portland-cement clinker to produce blended cements have themselves been used as admixtures to improve the sulfate resistance of concrete. In an investigation of portland blast-furnace slag cements, it was found [11] that, when tested by the procedure in which mortar bars are made with added sulfate and observed for length change during water storage, [15] the indicated sulfate resistance appeared to be primarily influenced by the calculated C_3A content of the portland-cement-clinker constituent of the blended cement (fig. 1). In this figure all of the curves show a tendency to rise and then level off but that the rise occurs at quite different ages, apparently not directly related to the C_3A contents or to the magnitude of the expansion. The curves show that one of the cements achieved 90% of its total expansion by an age of 21 days; three others achieved this by 84 days; two others by 112 days; while the remaining two did so only by 168 days age. These eight cements were also tested using the lean mortar-bar method(16) with the following comparative results:

Cement	Expansion at 1 Year, % x10^3	
	Lean Mortar Bars	Added-Sulfate Mortar Bars
341	420 (1)	748 (1)
339	386 (2)	436 (3)
345	292 (3)	295 (4)
340	166 (4)	690 (2)
342	75 (5)	203 (5)
337	72 (6)	162 (7)
338	56 (7)	172 (6)
336	24 (8)	89 (8)

The difference in position of cement 340 is noteworthy; by the lean mortar-bar method it is clearly fourth, and by the added-sulfate method it is just as clearly second; otherwise, the cements are ranked in substantially the same order by both testing procedures. In the lean mortar-bar test, the expansion began at approximately the same time and proceeded at essentially the same rate initally but subsided at varying rates proportional to ultimate expansion.

PORTLAND CEMENTS

Beginning in 1955 the Concrete Division undertook studies using the cements included in the Long-Time Study of Cement Performance in Concrete. Among the tests made was the mortar-bar test with added sulfate. The results obtained showed relationships similar to those previously found for the portland blast-furnace slag cements. The expansions at one year ranged from 1.374% to 0.073%. The relation between 28-day and 1-year expansion and calculated C_3A content is shown in fig. 2. The most striking anomaly in this relationship is in the data on cement 15 at the one-year age; this cement, with a calculated C_3A content of 12.5%, had an expansion at one year of only 0.132% or about what would be expected at 28 days for a cement of its C_3A content. Because of the peculiarity of this result, the test was repeated, and this time the result was substantially the same, 0.143%.

The results obtained with these cements again showed striking differences in the shapes of the curves for expansion versus time. Those for five of the cements are shown in fig. 3. For a number of cements, including cements 18, 12, and 13 as shown in fig. 3, the curve has the form of progressively increasing slope followed by a rather abrupt termination of expansion at ages as early as 28 days or as late as nearly a year. As previously noted, the test mortars are made with sulfate added to bring the SO_3 content to 7 percent of the weight of the cement, which is sufficient to convert 7.9% C_3A by weight of cement to high-sulfate calcium aluminum sulfate. Thus one might assume that the plateaus would all form at the same expansion level for all cements containing 7.0% or more SO_3 due to the sulfate having been used up and at progressively lower levels for cements of lower C_3A content. The observations do not bear out this explanation. The plateaus in fig. 3 are at the following levels:

Cement	C_3A, %	1-Year Expansion
18	13.4	1.374
12	12.6	0.399
31	11.0	0.262
51	3.8	0.073

Cement 13 at the 1-year age had shown an expansion of 0.408%, had shown no tendency to stop expansion, and had 10.3% C_3A.

Cement No. 15 has previously been noted as anomalous with respect to the relation of calculated C_3A content and observed expansion; cement 13 has an unusual shape for its time vs expansion curve and a high expansion for its C_3A content. Selected data for these two cements are given in table 3. These indicate that cement 15, with 2.5% more C_3A than cement 13 had only a third as much expansion. They were essentially of the same fineness but differed markedly in C_3S content, heat of hydration, and strength. The microscope observations of constituents probably consisting largely of material regarded chemically as "C_3A" were also markedly different. It is suggested that the slow rate of expansion in the sulfate resistance test of bars made with cement 13 is due to the fact that most of its "C_3A" was present as material identified microscopically as glass; that the low expansion of cement 15 was contributed to by its rapid rate of gain in strength and rigidity, and the high expansion of cement 13 was contributed to by its slow rate of gain in strength and rigidity. Blaine, Arni, and Evans[1] have found that for cements having high calculated percentages of C_3A, low expansions were associated with those of high Fe_2O_3 and CaO/SiO_2 ratio. Cements 13 and 15 did not differ significantly in Fe_2O_3 but cement 15, characterized by low expansion, had a high C_3S content and hence a high CaO/SiO_2 ratio. They also found from analysis of their data that cements with high compressive strength were associated with low expansion.

CURRENT STUDIES

Considerations of the sort previously mentioned have contributed to a belief that additional study is needed to evaluate the factors other than calculated C_3A content that influence the degree to which the cement contributes sulfate resistance to concrete. An investigation is now in progress using three cements having calculated C_3A contents of 5, 9, and 12% in mortars of eleven different compositions:

Cement/Sand Ratio	Water/Cement Ratio
2.0	0.3, 0.4, 0.5, 0.6
2.75	0.4, 0.5, 0.6
3.5	0.4, 0.5, 0.6, 0.7

Bars of these mortars are being stored in 5% sodium sulfate solution to an age of one year and observed periodically for development of changes in length, modulus of elasticity, porosity, and compressive strength. The data are being statistically analyzed. Other studies concerned with the composition and crystallinity of the alumina-containing phases in portland cement are also in progress.

CONCLUDING STATEMENT

I believe we know how to make portland-cement concrete that will successfully resist sulfate attack in the various environments in which we are called upon to place the concrete, but I am sure we do not know how to do this with a very high degree of efficiency and economy in the selection and use of cements. Progress is being made and work is under way that will further elucidate the influence of the several factors that interact to control sulfate attack and sulfate resistance. Only as these fundamental interactions are understood will we be able to use materials efficiently and economically to produce the desired degree of sulfate resistance with assurance.

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Table 1

Data on 19 Cements[8][9]

Sample No.	C_3A, %		X-ray Intensity, Counts/Sec (Peak Height - Background at 2.70 A)
	Micrometric	Calculated	
E-6	0.1	4.5	(a)
E-9	0.3	4.6	(a)
E-41	0.7	8.9	(a)
E-15	2.0	3.9 or 7.4	120[a]*
E-60	3.7	7.2	160[a]*
E-37	3.7	6.8	155[a]*
RC-318	4.4	6.2	165[a]*
E-33	4.8	10.0	210
RC-304	4.8	8.0	130
RC-299	5.4	12.6	245
RC-306	6.3	14.0	225
E-3	6.3	13.3	235
E-63	6.6	9.8	235
RC 305	6.9	10.6	225
RC 315	7.6	11.3	230
RC 300	8.7	13.4	245
RC 303	9.8	12.6	290
RC 316	10.0	11.2	250
E 58	17.4	17.3	450

(a) Peak too low and too poorly resolved for intensity measurement.
* In work reported earlier[8] no intensity measurement made; intensities measured later.[9]

Table 2

Data on 10 Cements[9]

Cement Serial No.	C_3A, %	MgO, %	C_3A Estimated from X-ray Diffraction	Expansion, %, at 1 Yr of Mortar Bars with Added Sulfate
395	6	0.58	4-5	0.063
396	5	3.64	<2	0.116
397	6	0.92	n.f.	0.083
398	5	1.32	n.f.	0.083
399	5	1.32	<2	0.078
400	7	3.99	n.f.	0.129
401	6	2.70	<2	0.116
412	5	2.94	<2	0.097
402	3	1.54	<2	0.052
407	3	0.82	?	0.054

n.f. = not found; possible masked by 2.72-A interference.

Table 3

Selected Comparative Data, Cements 13 and 15

	Cement 13	Cement 15
Calculated C_3A, %	10.3	12.5
Calculated C_3S, %	56.9	67.7
Compressive strength, psi, 3d	1800	3315
7d	2465	4650
28d	3875	6140
Heat of hydration, cal/g, 28d	85	116
Fineness, a.p., sq cm/g	3485	3340
Microscope data(18)		
C_3A, %	1.6)	8.6)
"Dark prismatic," %	1.9)11.7	1.2)10.1
Glass, %	8.2)	0.3)
Expansion, mortar bars, %, 1 yr	0.408	0.132 0.143

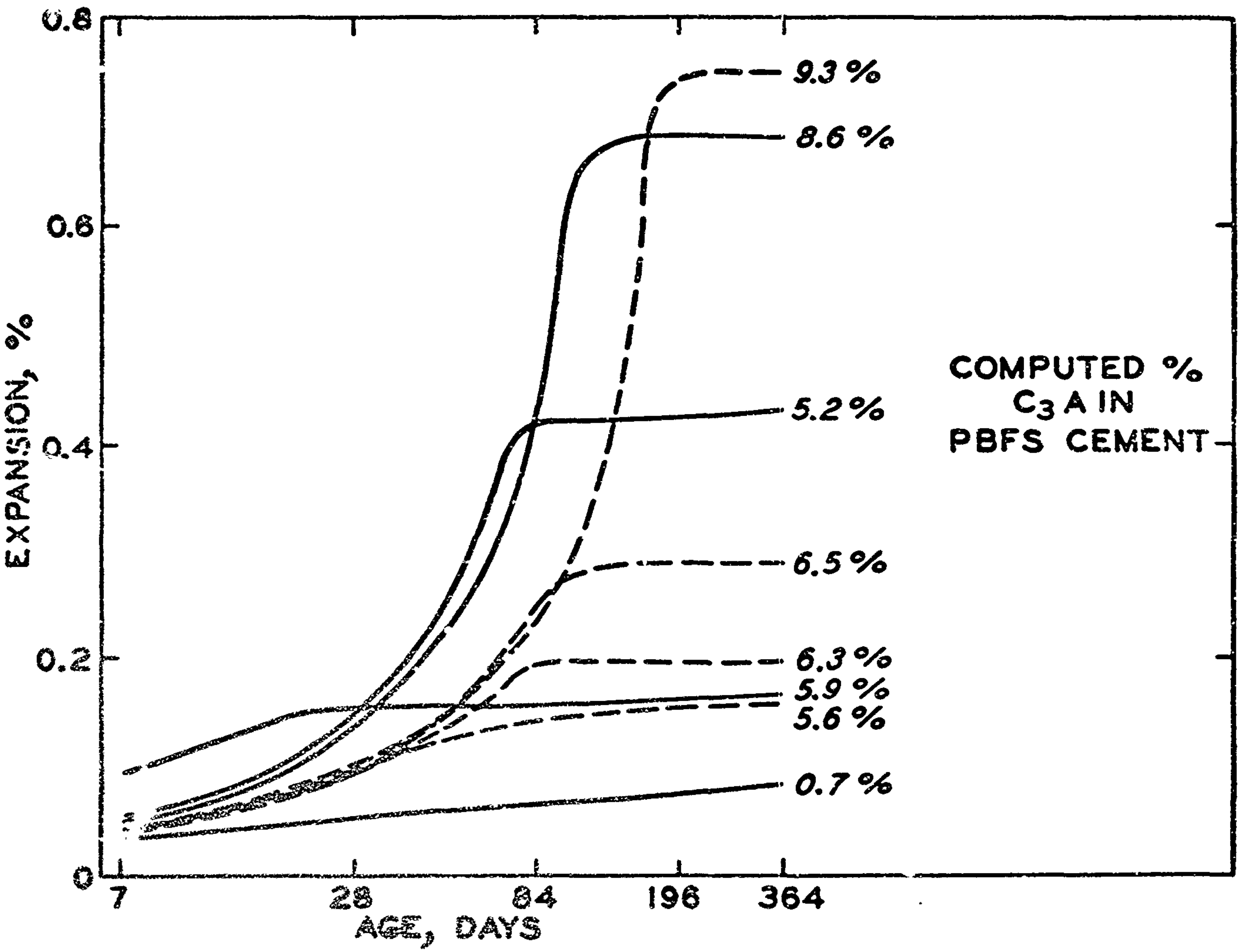

Fig. 1 Expansion of added-sulfate mortar bars made with portland blast-furnace slag cement.

Fig 1

4

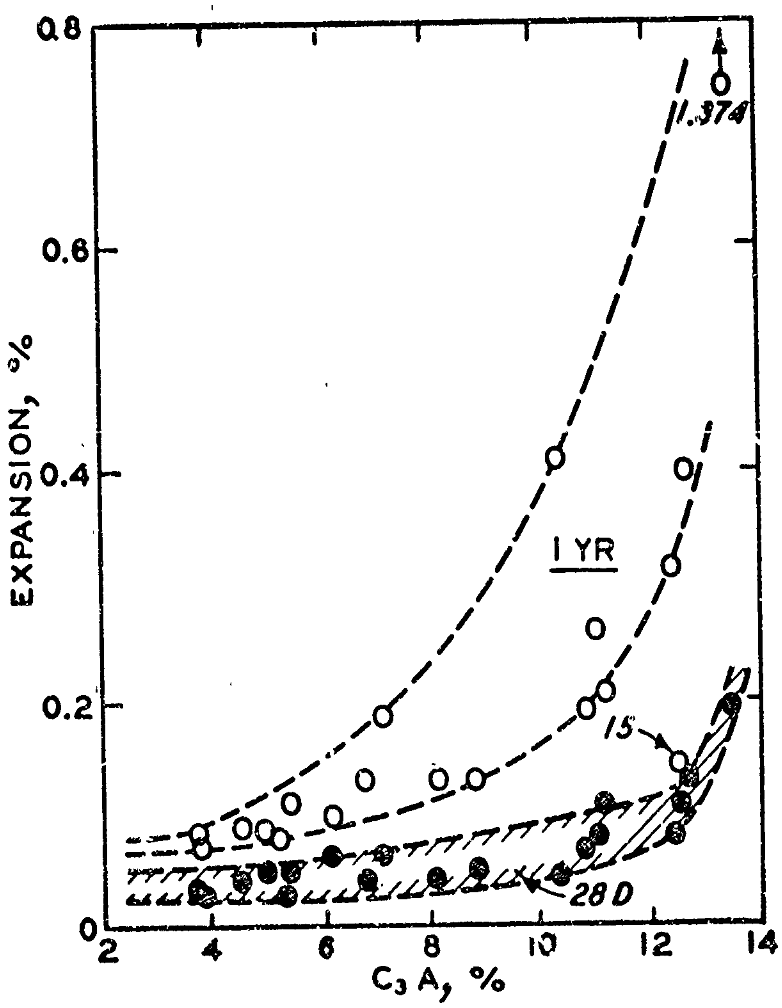

Fig. 2. Relation of expansion in sulfate resistance test to tricalcium aluminate content of cement

Fig 2

65

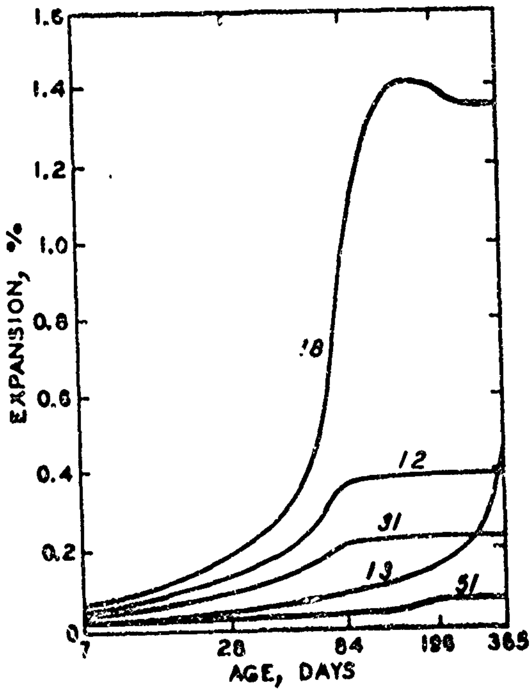

Fig. 3. Sulfate r(istance test results for selected cements